

TEMPERATURE DEPENDENCE ON THE STRENGTH AND STRESS RUPTURE BEHAVIOR OF A CARBON-FIBER REINFORCED SILICON CARBIDE MATRIX (C/SiC) COMPOSITE

Michael J. Verrilli and Anthony Calomino
NASA Glenn Research Center
21000 Brookpark Road
Cleveland, Ohio 44135

ABSTRACT

Tensile strengths and stress rupture lives of carbon-fiber reinforced silicon carbide (C/SiC) specimens were measured at 800 °C and are compared to previously reported 1200 °C data. All tests were conducted in an environmental chamber containing 1000 ppm of oxygen in argon. The average 800 °C tensile strength of 610 MPa is 10% greater than at 1200 °C. Average stress rupture lives at 800 °C were 2.5 times longer than those obtained at 1200 °C. The difference in the 800 and 1200 °C lives is related to the oxidation rate of the reinforcing carbon fibers, which is the primary damage mode of C/SiC composites in oxygen-containing environments.

INTRODUCTION

In order to significantly reduce the cost of placing payloads into orbit, NASA is developing advanced Reusable Launch Vehicles (RLVs) to replace the Space Shuttle. Carbon-fiber reinforced silicon carbide (C/SiC) composite is proposed for use in several high temperature propulsion and airframe structural components in these RLVs [1]. In support of the component design process, NASA Glenn is developing life prediction models for ceramic matrix composites, with a goal of accurate determination of the capability of C/SiC hardware for space propulsion applications [2]. Materials characterization is required to assess the strength and durability of C/SiC in support of this model development.

In this study, tensile strengths and stress rupture lives of C/SiC were obtained at 800 °C in order to generate a database for calibration of a Probabilistic Residual Strength (PRS) life prediction model [3]. The objective of this paper is to compare the 800°C behavior to previously reported 1200 °C data.

EXPERIMENTAL

The material tested was a woven carbon-fiber reinforced SiC matrix composite. The composite was fabricated by GE Power Systems Composites and

consisted of a CVI SiC matrix reinforced with a [0/90] plain weave T-300 carbon fiber cloth. Tensile and stress-rupture specimens, which had a 10.16 mm wide gage section, were machined from panels and then were seal coated with CVI SiC. More details of the C/SiC composite and test specimens can be found in reference 4.

Load-controlled tensile tests and stress-rupture tests were conducted at 800 °C. Sixteen tensile tests and sixteen stress-rupture tests were conducted. All testing was performed in an environment of 1000 ppm O₂ in argon, flowing at 0.5 liter per minute. This procedure was employed to avoid rapid oxidation of reinforcing carbon fibers that occurs when elevated temperature testing is conducted in air or oxygen [4-7]. These same test procedures were used to generate strength and rupture data at 1200 °C [4].

RESULTS

Figure 1 compares the strengths obtained from sixteen tensile tests of C/SiC specimens at 800 °C to the data set of twenty tests obtained at 1200 °C. The average 800 °C strength of 609.9 ± 37.4 MPa is 10 % greater than the average strength of 558.1 ± 29.6 MPa obtained at 1200 °C [4]. The average failure strain at 800 °C ($1.00 \pm 0.15\%$) was the same as obtained at 1200 °C ($0.951 \pm 0.10\%$).

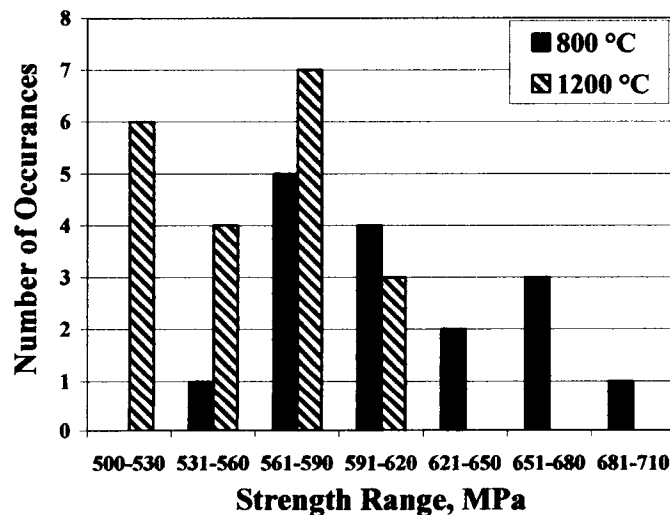


Figure 1 - Tensile strengths of C/SiC at 800 and 1200 °C.

The stress-rupture data is shown in Figure 2. Lives obtained at 800 °C were typically longer than at 1200 °C. The average life at 800 °C is about 2.5 times greater than that obtained at 1200 °C using the same stress. To aid in assessing the temperature dependence on rupture life, empirical curve fits were performed using the relationship

$$\sigma = m \cdot \log(t_r) + b \quad (1)$$

where

σ is the composite stress, t_r is the rupture time and b and m are constants. The fits of the 800 and 1200°C rupture data to Equation 1 are shown in Fig. 2. The empirical fits for the two temperatures have a similar slope.

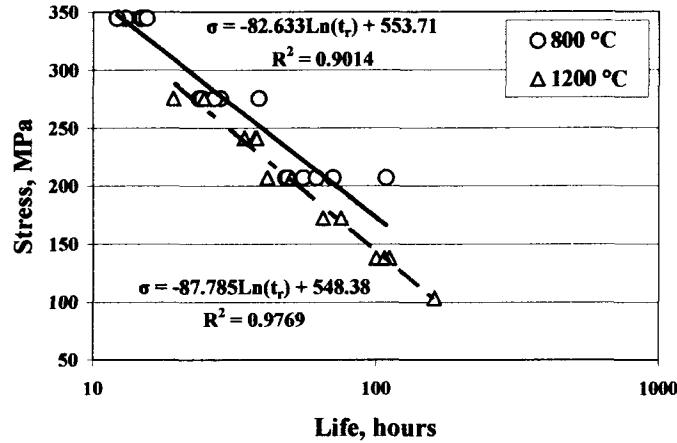


Figure 2 - Stress rupture data for C/SiC obtained at 800 and 1200 °C in 1000 ppm O₂/Ar.

From the strain versus time data obtained during the rupture tests, the secondary or minimum creep rate was determined. A plot of the minimum creep rate data versus rupture life for each temperature is shown in Figure 3. An empirical relationship similar to Eq. 1 was used to fit this data:

$$t_r = n \cdot \log(d\epsilon/dt) + c \quad (2)$$

where $d\epsilon/dt$ is the minimum creep rate and n and c are constants. The fits are

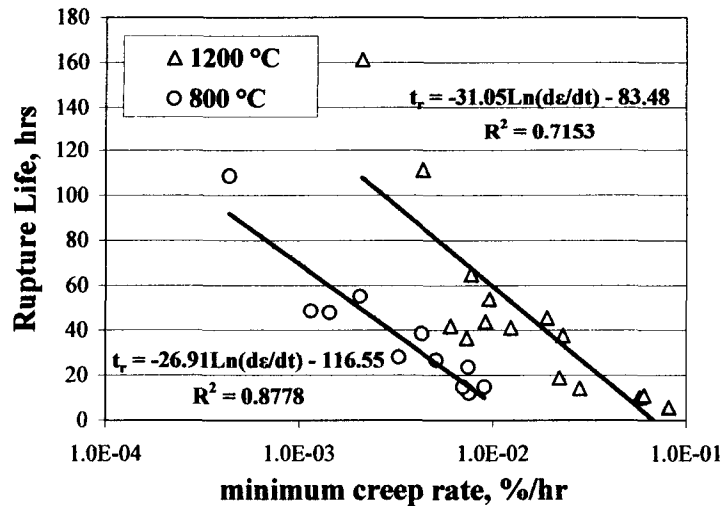


Figure 3 - Minimum creep rate data for C/SiC obtained at 800 and 1200 °C in 1000 ppm O₂/Ar.

shown in Fig. 3. For a given rupture life, the minimum creep rate at 1200 °C is about 10 times greater than at 800 °C.

Figure 4 shows polished cross-sections of specimens that were tested under stress-rupture conditions at 800 and 1200 °C in 1000 ppm O₂/Ar. Pores and consumed carbon fibers appear as black regions and the intact fibers and SiC matrix appear gray. In both specimens, extensive fiber oxidation can be seen in the tows adjacent to the composite surface. More extensive damage occurred in the specimen tested at 1200 °C, which resulted in spallation of material. Details of the oxidation at the mid-thickness of these specimens are shown in Figure 5. Fiber oxidation can be seen in the 800 °C specimen in fibers oriented both 0 and 90° to the load direction. No detectable carbon oxidation is visible in the 1200 °C specimen.

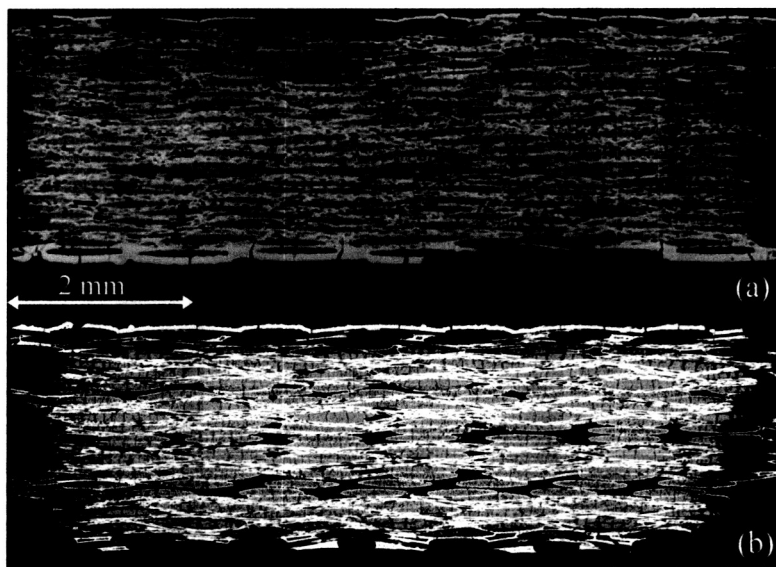


Figure 4 – Polished cross sections of stress-rupture tested C/SiC specimens, a) 800 °C, b) 1200 °C.

DISCUSSION

The damage modes found in the specimens tested in 1000 ppm O₂/Ar are the same as occurs in C/SiC tested in higher partial pressures of oxygen. At 1200 °C in air and in oxygen, the fiber oxidation kinetics under stress-rupture conditions result in a distinct reaction front of receding carbon [5]. Carbon and oxygen reactions occur very quickly, resulting in consumption of fibers around the periphery of the C/SiC specimens and little fiber oxidation in the interior of composite [8]. No visible fiber oxidation can be seen in the middle ply of the specimen tested at 1200 °C in 1000 ppm O₂/Ar (Fig. 5b). Significant oxidation of the outer fiber tows occurs as well at 800 °C in air and oxygen, but extensive carbon fiber oxidation also occurs throughout the specimen cross section (Fig. 5a). The same type of damage is visible in Figure 4a. Cracks in the as-fabricated

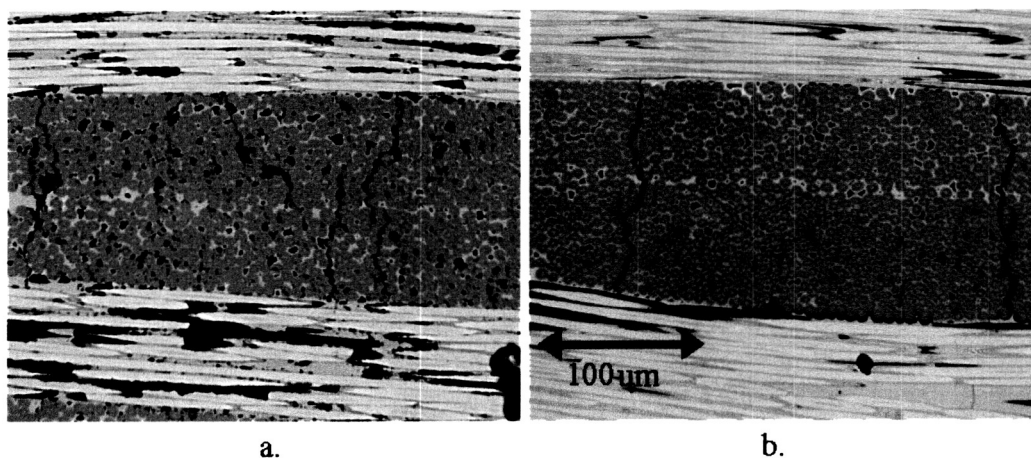


Figure 5 - Micrographs of specimens ruptured tested in 1000 ppm O₂/Ar, a) 800 °C, b) 1200 °C. Load direction is perpendicular to the images.

composite are the locations of fiber oxidation in the interior of the C/SiC specimens. ✓

The slopes of the stress versus life data for the two temperatures are similar (Fig. 2), as are the slopes of the minimum creep rates versus life (Fig. 3). This is consistent with the fact that the composite degradation mechanism of carbon fiber oxidation is occurring at different rates for these two temperatures due to the change in kinetics. Lower minimum creep rates and longer lives at 800 than at 1200 °C are in agreement with relative fiber oxidation rates predicted for C/SiC stress-rupture tested in air [8]. Note that time-dependent deformation of C/SiC in air is due to loss of carbon fibers through oxidation [4, 9].

Specimens tested under rupture conditions in air at 1200 °C typically failed outside of the gage length [5]. All but one of the specimens tested in this study at 800 °C and the specimens tested in the previous effort at 1200 °C [4] failed in the gage section, so the minimum creep rate data is reflective of damage rate that lead to specimen failure.

SUMMARY

The mechanical behavior of a C/SiC composite was characterized at 800 °C. Tensile strengths and rupture lives were generated in a low partial pressure of oxygen. This data was compared with strengths and rupture lives obtained at 1200 °C in a previous effort.

At a given test stress, the average lives obtained at 800 °C were about 2.5 longer than obtained at 1200 °C. For specimens that had the same life, the minimum creep rate at 800 °C was about 10 times slower than at 1200 °C. These results are consistent with observed temperature-dependent differences in composite damage through carbon fiber oxidation.

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REFERENCES

1. Kiser, J. D., Effinger, M. R., Glass, D. E., et al, "CMC Component Development Supporting the NASA-Led 2nd and 3rd Generation RLV Investment Areas: Propulsion Applications", in the proceedings of 25th Annual Conference on Composites, Materials, and Structures, Cape Canaveral, FL. January 22-25, 2001.
2. Levine, S., Verrilli, M. J., Calomino, A., Thomas, D. J., Halbig, M. C., Ellis, J. R., and Opilla, E. J., "Ceramics Matrix Composites (CMC) Life Prediction Development", in the proceedings of the 26th Annual Conference on Composites, Materials, & Structures, Cape Canaveral, FL, Jan. 28-31, 2002.
3. Thomas, D. J., Verrilli, M. J., and Calomino, A., "Stress/Life Behavior of a C/SiC Composite in a Low Partial Pressure of Oxygen Environment Part III: Life Prediction Using Probabilistic Residual Strength Rupture Model", Ceramic Eng. and Science Proceedings, vol. 23, issue 3, pp. 453 – 460, 2002.
4. Verrilli, M. J., Calomino, A., and Thomas, D. J., "Stress/Life Behavior of a C/SiC Composite in a Low Partial Pressure of Oxygen Environment, Part I – Static Strength and Stress Rupture Database", Ceramic Engineering and Science Proceedings, Vol. 23, No. 3, pp. 453- 442, 2002.
5. Halbig, M. C., "The Influence of Temperature, Stress, and Environment on the Oxidation and Life of C/SiC Composites", Ceramic Engineering and Science Proceedings, Vol. 23, No. 3, pp. 419- 426, 2002.
6. Lamouroux, F. and Camus, G., "Kinetics and Mechanics of Oxidation of 2D Woven C/SiC Composites: I, Experimental Approach," Journal of the American Ceramic Society, Vol. 77, No. 8, 1994, pp. 2049-2057.
7. Halbig, M. C., Brewer, D. N., and Eckel, A. J., "Degradation of Continuous Fiber Ceramic Matrix Composites Under Constant-Load Conditions", *Mechanical, Thermal, and Environmental Testing and Performance of Ceramic Composites and Components*, ASTM STP 1392, M. G. Jenkins, E. Lara-Curzio, and S. T. Gonzy, Eds. American Society for Testing and Materials, West Conshohocken, PA., pp. 290-304, 2000.
8. Halbig, M. C., "Modeling the Oxidation of Carbon Fibers in a C/SiC Composite under Stress Oxidation", Ceramic Engineering and Science Proceedings, Vol. 23, No. 3, pp. 427- 434, 2002.
9. Lara-Curzio, E., and Singh, M., "The Time-Dependent Deformation of Carbon Fiber Reinforced Melt-Infiltrated Silicon Carbide Composites: Stress-Rupture and Stress-Relaxation Behavior at 1000 °C", *Mechanical, Thermal, and Environmental Testing and Performance of Ceramic Composites and Components*, ASTM STP 1392, M. G. Jenkins, E. Lara-Curzio, and S. T. Gonzy, Eds. American Society for Testing and Materials, West Conshohocken, PA., pp. 216-228, 2000.

